DEVICE AND METHOD FOR CONNECTING INERT ANODES DESIGNED FOR THE PRODUCTION OF ALUMINIUM BY FUSED BATH ELECTROLYSIS

Field of the invention

The invention relates to the production of aluminium by fused bath electrolysis. It particularly concerns anodes used for this production and the electrical connection of these anodes to current input conductors.

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State of prior art

Metal aluminium is produced industrially by fused bath electrolysis, namely by electrolysis of alumina in solution in a bath based on molten cryolite, called an electrolytic bath, particularly using the well-known Hall-Héroult process. The electrolysis is done in cells comprising a crucible made of a refractory material capable of containing the electrolyte, at least one cathode and at least one anode.

The electrolysis current that circulates in the electrolyte through the anodes and cathodes causes alumina reduction reactions and is also capable of maintaining the electrolyte bath at the target operating temperature, typically of the order of 950°C, by the Joule effect. The electrolysis cell is regularly supplied with alumina so as to compensate for consumption of alumina caused by electrolysis reactions.

In the standard technology, anodes are made of a carbonaceous material and are consumed by aluminium reduction reactions. Consumption of the carbonaceous material releases large quantities of carbon dioxide.

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Aluminium producers have been searching for anodes made of non-consumable materials, called "inert anodes", for several decades, to avoid environmental problems and costs associated with manufacturing and use of anodes made of carbonaceous material. Several materials have been proposed, particularly ceramic materials (such as sno_2 and ferrites), metallic materials and composite materials such as materials known as "cermets" containing a ceramic phase and a metallic phase, particularly nickel ferrites containing a metallic copper-based phase.

Problems encountered in the development of inert anodes for the production of aluminium by electrolysis lie not only in the choice and manufacturing of the material from which the anode is made, but also in the electrical connection between each anode and the conductor(s) that will be used for the electrical power supply of the electrolytic cell. Several methods and devices have been proposed for the connection of inert anodes.

US Patent No. 4 500 406 proposes to use anodes with an active part, a metallic part suitable for connection, and a composition gradient between the active part and the metallic part. US Patent No. 4 541 912 describes an assembly formed by hot isostatic compression of a cermet material on a metallic conducting substrate. These

solutions make it more difficult to make the anode and impose constraints on baking parameters for the active part of the anode.

American patent US 4 623 555 describes the formation of a connection using a composition gradient formed by plasma sputtering. This solution requires perfect control of the process for formation of the intermediate layer and imposes a complex additional step.

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Patents US 4 468 298, US 4 468 299 and US 4 468 300 describe joints formed by diffusion, friction or other welding. Patent US 4 457 811 describes a connection comprising one or several elastic strips welded on the inner or outer surface of an anode. These solutions require a chemical reduction of the contact surface before formation of the joints, considerably complicating manufacturing of the anodes. Another disadvantage of these solutions is that they complicate the assembly of the electrical connections.

American patents US 4 357 226 and US 4 840 718 describe mechanical connections applicable to solid anode assemblies. These connection methods are complex.

US 4 456 517, US 4 450 061, American patents US 6 264 810 describe mechanical us 4 609 249 and connections applicable to anodes with a central cavity. These connections are sensitive to changes in 25 mechanical properties of its constituent elements when the anodes are used, and introduce mechanical tensions between the anode and the metallic parts. these solutions are sensitive to the corrosive ambient atmosphere of the electrolytic cells. In order to overcome this difficulty, some of these patents also propose to add screens and / or inert filling materials. These complementary protection means complicate the manufacture of connections and make it more expensive. The solution proposed in patent US 6 264 810 has the additional disadvantage that it requires a large number of distinct parts that must maintain their mechanical characteristics over a long period of time.

Therefore, the applicant searched for solutions to overcome the disadvantages of prior art.

Description of the invention

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An object of the invention is an anode assembly comprising at least one inert anode and at least one connection conductor intended for the electrical power supply of the anode, characterized in that:

- the anode is hollow and is in the form of a ladle,
- the contact surface between the conductor and the anode is close to the aperture of the anode (typically near the periphery),
 - the electrical and mechanical link between the conductor and the anode comprises a brazed metal joint that could be formed by fully or partly brazing during use.

In one advantageous embodiment of the invention, the said brazed joint could be consolidated during use of the said assembly in an electrolytic aluminium production

cell. It advantageously achieves this by including at least one element chosen from among aluminium, silver, copper, magnesium, manganese, titanium and zinc.

The anode is typically in the shape of a cylindrical ladle or a "glove finger", for which the outer surface of the closed end is rounded or is a rounded quadrangle in which the corners of the outer surface of the closed end are rounded. These shapes avoid disparities of local current density during use, when the closed end is immersed in an electrolyte bath based on molten salt.

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The applicant has noted that known connection modes that carry electrical power directly to the centre or close to the part immersed in the bath, entrain poor distribution of current lines, particularly in anodes in the shape of a ladle. The applicant has also noted that this distribution of current lines could lead to current densities that are too low at some locations (in other words typically less than about 0.5 A/cm²), which facilitates local corrosion, and is too high (in other words typically more than 1.5 A/cm², or even more than 2.5 A/cm²) at other locations, which locally accelerates degradation by electrochemical dissolution.

The applicant had the idea of using a brazed joint that increases in strength during a heat treatment, either (wholly or partly) before use of the assembly in an electrolytic cell, or (wholly or partly) in situ during use of the assembly in an electrolytic cell. The brazed joint avoids applying a mechanical tension on part of the inert anode used for the mechanical connection.

The brazed joint results in a common and efficient mechanical and electrical connection, which considerably simplifies the manufacturing process. This variant is also advantageous due to the fact that it enables the use of a mechanical assembly that is sized so that it is sufficient to temporarily and satisfactorily hold the anode in place mechanically until the brazed joint has gained strength, but is not necessarily capable of satisfying all mechanical needs of the connection required during use, since the increase in strength of the brazed joint provides the additional mechanical strength required in use.

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Another object of the invention is a method for manufacturing anode assemblies according to the invention.

Another object of the invention is the use of at least one anode assembly according to the invention, or obtained by the manufacturing process according to the invention, for the production of aluminium by fused bath electrolysis.

Another object of the invention is a cell for producing aluminium by fused bath electrolysis comprising at least one anode assembly according to the invention or obtained by the manufacturing process according to the invention.

The invention will be better understood after reading the detailed description of particular embodiments and the attached figures.

Figures 1 to 7 relate to the invention. Figures 1 and 3 to 6 illustrate anode assemblies according to the invention, seen in a longitudinal section. Figure 2 illustrates two elements of the anode assembly in Figure 1. Figure 7 illustrates the morphological change of the brazing material during brazing.

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The anode assembly according to the invention comprises at least one hollow inert anode (2), at least one connection conductor (3, 4, 4', 5) and at least one brazed metallic joint, or joint that could be formed by brazing, (31) capable of providing a mechanical and electrical connection (30) between the conductor and the anode.

The hollow shape of the anode limits the manufacturing cost and releases a useful space (21) inside the anode. For example, this space or cavity (21) may be used to put in one or several heating resistances (9) that can be used to heat the anode before it is immersed in the liquid electrolyte bath.

The anode has an inner surface (210) and an outer surface (230). The thickness E of the wall (23) of the anode may be different at different locations of the anode. The thickness of the lateral part (23') of the wall (23) of the anode may or may not be uniform.

In one particular embodiment of the invention, the anodes and the connection conductors are axially symmetric about a central axis A.

The closed end (24) of the anode (2) has a so-called "active" surface (240) that will be immersed into an

electrolyte bath based on molten salt. The active surface (240) of the anode is preferably free of sharp corners in order to prevent point effects in the distribution of the electrical current during use: it may be hemispherical or it may include polygons with rounded corners.

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According to the invention, the open end (22) of the anode (2) that is opposite the closed end (24) is used to make a mechanical and electrical connection to at least one connection conductor (3, 4, 4', 5). The joint (31) is at the connection area (25) of the anode.

More precisely, the anode assembly (1) to be used in a fused bath electrolysis aluminium production cell according to the invention comprises:

- at least one inert anode (2) in the shape of a ladle, with length L, comprising a cavity (21), an open end (22) comprising an opening (200), a wall (23) surrounding the cavity (21), a closed end (24) and at least one mechanical connection means (26, 27, 28, 29);
- at least one connection conductor (3, 4, 4', 5) comprising a connection end (42) and at least one mechanical connection (44, 45, 46) capable of cooperating with the mechanical connection means (26, 27, 28, 29) of the anode (2) so as to set up a mechanical link between the conductor and the anode;
 - at least one brazed metallic joint (31) or at least one brazing material that could form a brazed metallic joint (31) by brazing wholly or partly during use, the said joint (31) being located between all or

part of at least one surface (20, 20', 20") of the open end (22) of the anode (2) and all or part of at least one surface (40, 40', 40") of the connection end (42) of the conductor (3, 4, 4', 5).

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anode assembly elements Advantageously, the according to the invention, and particularly the said mechanical connection means (26, 27, 28, 29, 44, 45, 46), to be sufficient to provide sized so as may be satisfactory temporary mechanical support of the anode until the brazed joint has gained strength, before use or during use in an electrolytic cell.

The said joint (31) is located between all or part of at least one surface (20, 20', 20") of the open end (22) of the anode (2) and all or part of at least one surface (40, 40', 40") of the connection end (42) of the conductor (3, 4, 4', 5).

The connection conductor (3, 4, 4', 5) will be used to supply electrical power to the anode (2). It may comprise a central cavity (8). The connection conductor (3, 4, 4', 5) may be formed in several parts, and advantageously comprises at least one member (4) made of a nickel based alloy (in other words containing more than 50% by weight of nickel) and the connection end (42) is advantageously located on this member (4). The nickel based alloy is advantageously an UNS N06625 alloy called a "625 alloy" and is more advantageously an UNS N06025 alloy, called a "602 alloy", for which the content of added aluminium gives better resistance to corrosion when hot.

As illustrated in Figures 1, 3 and 4, the connection conductor (3, 4, 4', 5) may comprise an intermediate conductor (4), typically made of a nickel based alloy, designed to create a mechanical and electrical connection with the anode, and an "external" conductor (5) designed for the mechanical support of the anode assembly and electrical connection outside the electrolytic cell, usually by an external connection means (6). As illustrated in Figure 5, the connection conductor (3, 4, may comprise two or several intermediate conductors (4, 4'). The parts (3, 4, 4', 5) are fixed to each other by one or several intermediate connections **(7)**.

The shape of the connection conductor (3, 4, 4', 5) is typically elongated and possibly tubular.

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The mechanical connection means (26, 27, 28, 29) of the anode (2) is/are located close to the open end (22). They cover part of the open end (22) of the anode, typically representing less than 10% or even less than 5%, of the total length L of the anode.

In order to provide a sufficient electrical contact, the total area of the connection surface(s) (20, 20', 20") of the anode is such that the current density per unit area at the nominal intensity during use, is preferably between 1 and 50 A/cm², more preferably between 2 and 20 A/cm², and even more preferably between 5 and 15 A/cm². These areas are typically between 1 and 20%, or even between 5% and 15%, of the total area of the external surface (230) of the anode.

The mechanical connection means (26, 27, 28, 29) of the anode (2) typically comprise(s) at least one element chosen from among the collars (26), annular cavities (27), annular grooves (28) and annular shoulders (29). These shapes are easy to obtain on inert anodes with axial symmetry.

The mechanical connection means (44, 45, 46) of the conductor (3, 4, 4', 5) is/are preferably close to the connection end (42).

The mechanical connections means (44, 45, 46) of the conductor (3, 4, 4', 5) typically comprise(s) at least one element chosen from among annular grooves (44), skirts (45) and annular shoulders (46). These shapes are easy to obtain - typically by screw cutting - on mechanical parts with axial symmetry.

The anode connection means (26, 27, 28, 29) and the conductor connection means (44, 45, 46) advantageously cooperate through at least one of the means chosen among screwing, click fitting, friction, insertion or force fitting. Insertion and force fitting may be done after heating the anode and / or the connection conductor.

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The anode assembly (1) may comprise one or several complementary assembly means (34, 340, 36) such as one or more clamping rings (34, 340) and one or more open or closed rings (36).

The connection surfaces (20) close to the opening (200) of the anode (2) are advantageously inclined (typically from the assembly axis A) so as to prevent flow of the brazing material (31') in the cavity (21)

during brazing and / or use of the anode assembly. For that purpose, the connection surface(s) (20, 20', 20") of the anode (2) typically comprise at least one flat surface element (20) for which the tangent forms an angle α between 45° and 90°, or even between 60° and 90°, with the main axis A of the anode.

The connection surfaces (20, 20', 20") are typically at least partly on the external surface (230) of the anode (2) when the coefficient of expansion of the material from which the anode is made is less than the coefficient of expansion of the material from which the connection conductor is made; otherwise, they are typically at least partly on the inner surface (210) of the anode.

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15 The anode assembly (1) may also comprise at least one complementary seal (33) designed to confine the brazed joint (31), generally by limitation of the flow of the brazing material. This flow may take place during the heat treatment or during use. The complementary seal (33) is typically chosen from among open or closed rings and O-rings. The complementary seal (33) may be metallic or non-metallic.

Preferably, assembly of the conductor (3, 4, 4', 5) and the anode (2) does not involve any tightening or stress between the anode and the conductor, to limit the development of mechanical tensions before and / or during brazing.

Preferably, during use, the connection means (26, 27, 28, 29, 44, 45, 46) are located in a part of the cell

at least partially isolated from corrosive gases and at a temperature significantly lower than the bath temperature (and preferably less than 850° C), which is done by adaptation of the length L of the inert anode.

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In the embodiments illustrated in Figures 1, 3 and 5, the periphery of the opening (200) of the anode (2) comprises a collar (26) facing the outside of the anode and an annular cavity (27) also facing the outside of the anode. The connection conductor (3, 4, 5) comprises a skirt (45) threaded on the inside. The connection means also comprise a clamping ring (34) threaded on the outside and that can be screwed inside the skirt (45).

In the embodiment shown in Figure 1, the metallic joint (31) is formed from a brazing material in the form of a thin and flat ring, placed in the space (32) between the connection surfaces (20, 20") and (40, 40"). The connection means may comprise a ring (33) to limit the flow of the brazing material. Before the brazing operation, the threaded clamping ring (34) is screwed inside the skirt (45) so as to bring the connection surfaces (20, 20") and (40, 40") close to the brazing ring (31). The connection surfaces may possibly be put into contact with or may bear on the brazing ring.

As illustrated in Figures 3 to 5, the metallic joint (31) may be formed from a brazing material originating wholly or partly from at least one reservoir (35). The space (32, 32") is designed to accumulate brazing material and to form a joint (31) during brazing. The surface (20) close to the opening (200) is preferably

inclined so as to prevent the brazing material from flowing into the anode cavity (21).

In the embodiment shown in Figure 3, before the brazing operation, the threaded tightening ring (34) is screwed inside the skirt (45) so as to bring the connection surfaces (20, 20') and (40, 40') close to each other while leaving a space (32, 32') in which brazing material will accumulate, and to form a joint (31) during brazing.

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In the embodiment shown in Figure 4, the periphery 10 of the opening (200) of the anode (2) comprises annular groove (28) facing the outside of the anode. The connection conductor (3, 4, 5) comprises a skirt (45) provided with an annular groove (44) facing inwards. connection means also comprise a click fit ring 15 capable of cooperating with annular grooves (28) and (44) so as to set up a mechanical link between the conductor (4) and the anode (2). In these embodiments, the anode (2) is inserted inside the skirt (45) until click fitting into grooves (28) and (44) before the brazing operation. 20 There is a space (32) between the connection surfaces (20, 20') and (40, 40').

In the embodiment illustrated in Figure 5, the periphery of the opening (200) of the anode (2) comprises a collar (26) facing the outside of the anode and an annular cavity (27) also facing the outside of the anode. The connection conductor (3, 4, 4', 5) comprises a skirt (45) on which a clamping ring (340) can be fitted, typically using attachment means (37) such as bolts.

Before the brazing operation, the clamping ring (340) is fixed to the skirt (45) so as to trap the collar (26) while leaving a space (32, 32') designed to accumulate the brazing material and to form a joint (31) during brazing. The junction between the conductor (4) and the anode (2) remains loose until brazing.

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In the embodiments shown in Figures 1, 3 and 5, the connection means may comprise a ring (Figures 1 and 5) or a 0-ring (Figure 3) (33) to limit flow of the brazing material.

In the embodiment shown in Figure 6, the connection conductor (4) is provided with an annular shoulder (46) capable of cooperating with a corresponding annular shoulder (29) on the anode (2). The dimensions of these shoulders are such that the assembly can be made by hot expansion of one of the two parts: (A) when hot, the space G between the parts is large enough to enable the anode to be inserted into the conductor; (B) when cold, the shoulders are inserted one into the other to provide temporary mechanical support until the brazed joint (31) has increased in strength. The heating temperature for assembly is preferably lower than the melting temperature of the brazing material to prevent it from flowing during assembly.

As in the case of the configuration shown in Figure 6, the space (32') between some surfaces facing each other (20', 40') that will be brazed may be substantially vertical or conical.

The position and shape of the brazing material may change during brazing. Thus, as illustrated in Figure 7, the brazing material that has a determined initial shape and position (31') (Figure 7A) may deform during the heat treatment, typically by flowing, to occupy a final volume (31) in intimate contact with the connection surfaces (20, 20', 20", 40, 40', 40") (Figure 7B). The initial position may be wholly or partly in a reservoir (35).

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The anode assembly may comprise a thermal insulation (10) in the central cavity (21) of the anode, particularly in order to prevent overheating of the external connection conductor (5) due to internal radiation of the anode.

The anode (2) is typically chosen from among anodes comprising a ceramic material, anodes comprising a metallic material and anodes comprising a cermet material.

The manufacturing method for an anode assembly (1) according to the invention comprises:

- the supply of at least one inert anode (2) in the form of a ladle, with length L, comprising a cavity (21), an open end (22) comprising an opening (200), a wall (23) surrounding the cavity (21), a closed end (24) and at least one mechanical connection means (26, 27, 28, 25 29):
 - the supply of at least one connection conductor (3, 4, 4', 5) comprising a connection end (42), and at least one mechanical connection means (44, 45, 46) capable of cooperating with the mechanical connection

means (26, 27, 28, 29) of the anode (2) so as to set up a mechanical connection between the conductor and the anode;

- the supply of at least one brazing material capable of forming a metallic joint;
 - placement of the brazing material(s) at a determined location close to at least one of the surfaces (20, 20', 20") of the open end (22) of the anode (2) or the surfaces (40, 40', 40") of the connection end (42) of the conductor (3, 4, 4', 5) that will be connected by brazing;

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- assembly of the conductor (3, 4, 4', 5) and the anode (2) so as to bring the said surfaces (20, 20', 20", 40, 40', 40") close to each other;
- 15 a heat treatment capable of causing the formation of a brazed joint (31) between the conductor and the anode starting from the brazing material(s).

The brazed joint (31) is formed between the said surfaces (20, 20', 20", 40, 40', 40") and thus forms a mechanical and electrical connection between the conductor and the anode.

The assembly operation of the conductor (3, 4, 4', 5) and the anode (2) preferably produces a loose assembly, which will only become rigid during the heat treatment. This variant avoids mechanical stresses.

According to one advantageous embodiment of the invention, the composition of the brazing material, or one of the brazing materials, may be modified during the heat treatment so as to increase the melting temperature

up to a value greater than the maximum temperature applied to the said brazed joint (31) during use. This modification strengthens the joint. It may be obtained by at least one of the following mechanisms:

- 5 evaporation of at least one part of one of its constituent elements, the said element for example being zinc or magnesium;
 - chemical reaction of at least part of one of its said constituent elements with one of the constituents of the ambient atmosphere, particularly oxygen. For example, the said constituent element could be aluminium, zinc, magnesium or phosphorus;

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exchange by diffusion, with or without oxidation - reduction reaction, of at least one element with one of the said surfaces (20, 20', 20", 40, 40', The exchange may take place from the brazing material to the adjacent surface and / or from the adjacent surface to the brazing material. In the latter case, all or part of the said surfaces (20, 20', 20", 40, 40', 40") can be coated with a material comprising an element such as nickel, that can diffuse in the brazing The exchange can possibly take place by material. oxidation - reduction reactions. More precisely, the said composition may contain at least one element that could be exchanged by at least one oxidation - reduction reaction with the said inert anode (2), the said element typically being chosen from among magnesium, aluminium, or phosphorus, titanium, zirconium, hafnium or zinc.

mechanisms may be obtained with brazing materials chosen from among alloys or mixtures containing copper, silver, manganese and / or zinc.

The said surfaces (20, 20', 20", 40, 40', 40") may be fully or partly coated with a material that can be wetted by the brazing material(s).

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one advantageous variant of the According to invention, the brazing material(s) are wholly or partly inserted into the space that separates the surfaces (20, 20', 20") and (40, 40', 40") that will be brazed. other words, the said placement includes introduction of at least part of the brazing materials between all or part of at least one surface (20, 20', 20") of the open end (22) of the anode (2) and all or part of at least one surface (40, 40', 40") of the connection end (42) of the 15 conductor (3, 4, 4', 5).

According to another advantageous variant of the invention, the conductor (3, 4, 4', 5) includes at least one reservoir (35), the said placement includes the introduction of at least one brazing material into at least one reservoir (35) before the heat treatment, and the conductor (3, 4, 4', 5) and the anode (2) are assembled so as to leave a free space (32, 32') between the conductor and the anode. The brazing material(s) is (are) introduced between all or part of at least one surface (20, 20', 20") of the open end (22) of the anode (2) and all or part of at least one surface (40, 40', 40") of the connection end (42) of the conductor (3, 4,

4', 5) by flow of the said material during the heat treatment.

The heat treatment is advantageously performed while the anode assembly (1) is being used in an electrolytic cell.

The known connection modes are at the temperature of the immersed part of the anode and therefore close to the temperature of the electrolytic bath, while the connection according to the invention results in a very uniform temperature while maintaining the connection temperature equal to a value significantly lower than the electrolysis temperature, which reduces electrical, mechanical and chemical stresses on the connection.

15 Tests

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Test_1

A connection test was made with a device similar to that shown in Figure 5.

In this test, the anode was a cermet for which the ceramic phase comprised a nickel ferrite and the metallic phase was based on copper.

The brazing material was a CuZn alloy with 60% by weight of Cu and 40% by weight of Zn. The melting interval of this alloy was 870 to 900°C. The connection was preheated to 900°C before the anode was used in an electrolytic cell, for which the bath was based on molten cryolite. Partial melting of the brazing material at the time of preheating was sufficient to make a satisfactory

electrical connection. During disassembly, it was observed that the zinc was partly evaporated and oxidised and that use had made a complementary treatment necessary that increased the melting temperature of the joint well above 900°C.

Test 2

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A connection test was carried out with a device similar to that shown in Figure 6.

In this test, the anode was made of cermet with the same composition as in test 1.

The brazing material was a CuZn alloy with 30% by weight of Cu and 70% by weight of Zn. The melting interval of this alloy was 700 to 820°C. The brazing heat treatment was done entirely in situ. It resulted in a brazed joint offering an electrical connection stable in time and with a low electrical resistivity.

In tests 1 and 2, the outside diameter Do of the anode was typically of the order of 70 to 75% of the length L of the anode. The inside diameter D of the anode was also equal to about 60 to 65% of the outside diameter. The thickness E of the sidewall was uniform.

List of numeric marks

- 1. Anode assembly
- 2. Anode
- 3. Connection conductor
- 4. Intermediate connection conductor
- 5 4'. Intermediate connection conductor (extension)
 - 5. External connection conductor
 - 6. External connection means
 - 7. Intermediate connection
 - 8. Central cavity of the connection conductor
- 10 9. Heating resistance
 - 10. Thermal insulation
 - 20,20',20". Anode connection surface
 - 21. Anode cavity
 - 22. Open end
- 15 23. Anode wall
 - 23'. Side part of the anode wall
 - 24. Closed end of the anode
 - 25. Anode connection area
 - 26. Collar
- 20 27. Annular cavity
 - 28. Annular groove
 - 29. Annular shoulder
 - 30. Conductor / anode connection
 - 31. Brazed metallic joint
- 25 31'. Brazing material
 - 32, 32'. Space between connection surfaces of the anode and the conductor

- 33. Complementary seal
- 34. Threaded clamping ring
- 35. Reservoir
- 36. Ring
- 5 37. Attachment means
 - 40, 40', 40". Connection surface of the connection conductor
 - 41. Central cavity of the intermediate connection conductor
- 10 42. Connection end
 - 43. Wall of the intermediate connection conductor
 - 44. Annular groove
 - 45. Skirt
 - 46. Annular shoulder
- 15 200. Opening
 - 210. Inner surface of the anode
 - 230. Outer surface of the anode
 - 240. Active surface of the anode
 - 340. Clamping ring